AD NUMBER

AD832013

NEW LIMITATION CHANGE

TO

Approved for public release, distribution unlimited

FROM

Distribution authorized to U.S. Gov't. agencies and their contractors; Critical Technology; APR 1968. Other requests shall be referred to Air Force Aero Propulsion Lab., APIE-3, Wright-Patterson AFB, OH 45433.

AUTHORITY

Air Force Aero Propulsion Lab ltr dtd 12 Apr 1972

AD832013

300°C TRANSFORMER RECTIFIER

L. Krassner

RCA ELECTRONIC COMPONENTS

Somerville, New Jersey

INTERIM TECHNICAL REPORT NO. 1

April 1968

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Aero Propulsion Laboratory APIE-3.

Air Force Aero Propulsion Labortory APIE-3

Air Force Systems Command

Wright-Patterson Air Force Base, Ohio

300°C TRANSFORMER RECTIFIER

L. Krassner
RCA ELECTRONIC COMPONENTS
Somerville, New Jersey

INTERIM TECHNICAL REPORT NO. 1
April 1968

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Aero Propulsion Laboratory APIE-3.

Air Force Aero Propulsion Labortory APIE-3
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

FOREWORD

This interim technical report covers the work performed under Contract F33615-68-C-1245 from 1 January 1968 to 31 March 1968.

Dr. R. B. Janes, Manager, Technical Programs Laboratory, has overall supervision of the program. P. Gardner, Group Leader, Advanced Device Technology, is the project manager. A. Mayer, Group Leader, Advanced Device Chemistry, is principal investigator.

The contract with RCA Electronic Components, Somerville, New Jersey, was initiated under Project No. 3145 and is being administered under the technical direction of Mr. L. E. Schott of the Air Force Aero Propulsion Laboratory APIE-3, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

L. E. Schott
Project Engineer

ABSTRACT

Techniques for preparing gallium-arsenide surfaces free of damage and suitable for epitaxial growth have been investigated. A method of substrate preparation to be used for preliminary growth studies has been introduced. Growth studies have been started in a water vapor transport epitaxial system. This system has been constructed specifically to evaluate the structure of epitaxial films in relation to the substrate. Design improvements to reduce contamination of epitaxial layers due to leaks and corrosion have been made on the vapor hydride epitaxial system. This improved system is expected to be in operation shortly.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

SECTION I
OBJECTIVE

The goal of this program is the fabrication of 50- to 100-ampere rectifiers with reverse breakdown voltage of 150 volts, operable in a 300°C ambient temperature.

SECTION II
INTRODUCTION

The greatest problem encountered in the course of previous development work on high-temperature gallium-arsenide rectifiers (Air Force Contract 33 (615)-5352) was achieving consistent quality of epitaxially-grown gallium-arsenide material. To meet the increased performance goals of the present contract, it was deemed necessary to examine the vapor phase epitaxial growth process in great detail and to isolate and control all the variables that influence growth. Only, with thorough control of the epitaxial growth can material of adequate quality be obtained.

Consequently, effort has been concentrated on the improvement of epitaxial-growth systems and the preparation of substrates for epitaxy. Improvements were made in the design of the vapor hydride growth system to render it leakproof, and a system incorporating these changes is being assembled. In addition, a water vapor transport system for gallium arsenide epitaxial growth was assembled and will be used to evaluate substrate preparation and its effect on layer quality. It is expected that growth in the water system, with oxygen present, will provide information about the role of oxygen in gallium arsenide epitaxy, and its effect on the electrical properties of layers.

Epitaxial growth takes place on a polished substrate, and the influence of substrate properties on the grown layer are to be studied also. These properties include impurities, dislocations, and, in particular, damaged layers resulting from preparation and polishing of the substrate.

Using the information gathered from these growth studies, improvements in surface preparation and criteria for selection of substrates will be developed.

BLANK PAGE

SECTION III

PROGRESS OF WORK

A. SUBSTRATE MATERIALS

1. SAW DAMAGE

Preparation of substrate surfaces for epitaxial growth generally consists of two steps: lapping to remove saw damage, and polishing to produce a smooth, damage-free surface for growth. Inspection of the polished surface under differential interference microscopy is supplemented by preferential etching[1] to reveal damage caused by lapping and polishing operations, and dislocations.

Wafers to be used for growth substrates are sawed from ingots of gallium arsenide which are prepared by the horizontal gradient-freeze method. The ingot is oriented to provide either $\{\bar{1}\bar{1}\bar{1}\}$ or $\{100\}$ faces, and is cut with a stainless steel diamond-bonded saw blade. The surface of a sawed wafer has the appearance of A, Figure 1, in which the saw marks are visible.

Etching such a sawed wafer to remove 8 to 10 micrometers of material leaves the surface shown in B, Figure 1. In comparing B, Figure 1 to A, Figure 1, note that the ridges, which are artifacts of sawing, are more widely spaced than the original saw marks. A photomicrograph of a sawed wafer that has been etched with a preferential etch to reveal dislocations is shown in C, Figure 1. The dark elongated pits are similar in character to dislocations delineated on $\{100\}$ gallium-arsenide faces. These pits are aligned parallel (more or less) to the background of saw marks. A comparison of the three figures indicates that sawing leaves a highly damaged layer characterized by the closely spaced ridges of A, Figure 1. Removal of about 10 micrometers eliminates much of the saw-damaged material.

2. LAPPING

To produce a flat substrate surface for growth, and to remove all

A. SAWED GaAs SLICE, {100} FACE (X130)

B. SAWED AND ETCHED (1% Br_2) GaAs SURFACE (X130)

NOTE: APPROXIMATELY 10 μM REMOVED {100} FACE

C. SAWED GaAs SLICE AFTER DISLOCATION ETCH, {100} FACE (X130)

01884P

Figure 1. Photomicrographs of Wafer Surfaces

saw damage, it is desirable to lap away the saw damage and then to obtain a final polished surface with minimum damage. To this end, several lapping compounds were evaluated. Because the subsurface damage induced by a lapping procedure is related to the particle size of the lapping compound, rather fine compounds were used. The smallest sizes available in garnet and alumina abrasives were tested. Table I summarizes lapping compound particle size and evaluates each compound tested.

TABLE I

Compound	Size* (micrometers)	Quality of Surface
W-12 Garnet	4	Matte finish when >50 micrometers of material are removed. When lapped on Pellon paper, less surface roughness.
W-14 Garnet	3	Matte finish when >50 micrometers of material removed. Depth of irregularities ~0.5 micrometer.
W-16 Garnet	2	Semipolished but scratched appearance with up to 100 micrometers lapped.
Linde A α-alumina	0.3	Polished but scratched appearance. Very slow rate of material removal.
AC-DO3 unfused synthetic alumina, platelet form	3	Shallow scratches, least surface irregularities.

* Size refers to the most common particle size present in the grade described. A residual distribution of particles up to four times the given size is normally present, and may be considered the cause of the deeper scratches.

The criteria for evaluating the results of the various laps were microscopic examination, preferential etching, and Talysurf profiles of surface roughness. Examples of all these evaluations are provided in Figures 2 to 4. Table I contains a summary of results for all the procedures used.

From the Talysurf traces of Figure 3, it is evident that the degree of surface roughness is reduced by lapping on a paper-backed wheel rather than directly against the steel plate. The damage present in scratches causes

A. GaAs SURFACE LAPPED WITH W-12 GARNET TO REMOVE 100 MICRONS OF MATERIAL (A MATTE FINISH IS OBTAINED) (X64)

B. SURFACE PREPARATION OF A, FIGURE 2 AFTER PREFERENTIAL ETCHING TO REVEAL DAMAGE (X64)

01885P

Figure 2. Microscopic Evaluation of W-12 Garnet Lapped Surface

A. TALYSURF TRACE OF SURFACE DEPICTED ON A, FIGURE 2 (THIS SURFACE WAS LAPPED ON A PELLON PAPER BACKED STEEL PLATE)

SCALE: VERTICAL: 1 DIVISION = .127 μM
HORIZONTAL: 1 DIVISION = 50 μM

B. TALYSURF TRACE OF GaAs SURFACE LAPPED WITH W-12 GARNET AGAINST STEEL PLATE

SCALE: VERTICAL: 1 DIVISION = .254 μM
HORIZONTAL: 1 DIVISION = 50 μM

01886P

Figure 3. Talysurf Profiles of Surface Roughness

A. GaAs SURFACE LAPPED WITH 3 MICRON PLATELET ALUMINA, 56 μM REMOVED (X130)

B. SURFACE OF A, FIGURE 4 AFTER DIFFERENTIAL ETCH TO REVEAL DAMAGED REGIONS (X130)

C. TALYSURF TRACE OF GaAs SURFACE SHOWN IN A, FIGURE 4. THE OVERALL SLOPE OF THE TRACE IS CAUSED BY THE CONVEX WAFER SURFACE

SCALE: VERTICAL: 1 DIVISION = 254 Å
HORIZONTAL: 1 DIVISION = 50 μM

01867P

Figure 4. Evaluation of Platelet Alumina Lapped Surface

rapid etching of certain planes exposed in the scratch. In Figure 4, note that the etch figures generated in the scratches are oriented parallel to those which are caused by dislocations, i.e., outside scratches.

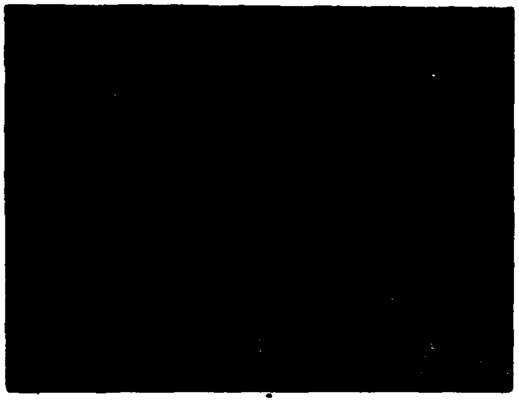
Platelet alumina is especially recommended to minimize subsurface damage. The contrast in Talysurf traces between the depth of scratches generated by the alumina and those generated by equivalent lapping with W-14 garnet is striking evidence of its superiority. From this evaluation, it was determined that lapping would be carried out on a Pellon paper-backed wheel using platelet alumina. Removing 50 micrometers of material was judged to be optimum on the basis that this is sufficient to remove saw samage without introducing a buildup of gallium arsenide in the Pellon backing paper.

3. POLISHING

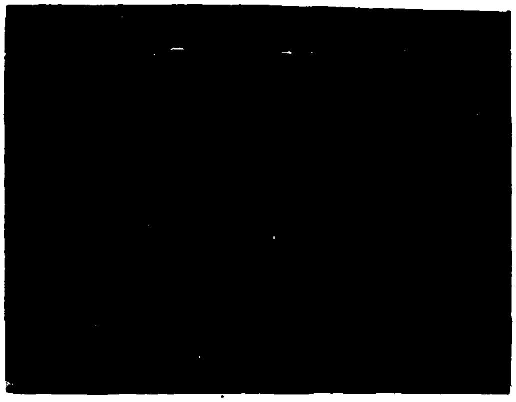
It is desirable to have both a flat, mirror-smooth surface, such as can be produced by mechanical polishing, and a damage-free surface, which results from etching. Etching alone generally produces a rippled, "orange peel" surface which will be propagated through the epitaxial growth and results in nonplanar junctions and an uneven surface for contacting. A mechanical polish causes some work damage no matter how fine the polishing abrasive.

The best compromise was obtained with a chemical polishing operation using 1 percent bromine in methanol solution. Chemical polishing is performed on a lapping machine with the wafers weighted in contact with Pellon paper, but without any abrasive material. Photomicrographs of the surface obtained by this method and of damage revealed by preferential etching and Talysurf traces are included in Figure 5.

At present, the surface prepared with platelet-alumina abrasive and bromine-methanol etching is considered to be the best that can be used as an epitaxy substrate. Bromine methanol is preferred to other potential etches both because it has been found to give particularly smooth, polished surfaces[(2)], and because there is no potential doping element in its constituents. Improvements to the polishing procedures will be made based on the results of the epitaxial growth.



A. ONE MINUTE PREFERENTIAL ETCH OF SURFACE TO REVEAL DAMAGE (X130)



B. FIVE MINUTE PREFERENTIAL ETCH OF POLISHED GALLIUM ARSENIDE WAFER (X130)

SCALE:

VERTICAL:
1 DIVISION = 254 Å

HORIZONTAL:
1 DIVISION = 50 μM

C. TALYSURF TRACE OF POLISHED GALLIUM ARSENIDE SURFACE. OVERALL CURVATURE IS CONCAVITY OF THE WAFER

Figure 5. Evaluation of Chemically Polished Surfaces

B. EPITAXIAL GROWTH SYSTEMS

1. WATER VAPOR TRANSPORT EPITAXY

A water vapor transport system for gallium-arsenide growth has been designed and constructed. The system is similar to that described by Lawley[3]. Its primary purpose is as an investigative tool, and not to prepare material for the eventual rectifier. Because the growth cycle in the water vapor transport system is more rapid than in the vapor hydride process, the water vapor transport system is useful for evaluating the structural properties of grown layers as related to substrate structure and its preparation.

Since the water vapor transport system necessarily involves the presence of oxygen, it is also expected to provide information on the influence of oxygen in epitaxially-grown material. Some comparison with the hydride system, which is designed especially to exclude oxygen leaks, will be possible.

The operation of the water vapor transport system can be described with the aid of Figure 6. The objective is the transport of gallium arsenide from the source material by water vapor, and re-formation of epitaxially-grown gallium arsenide on the substrate. The steps involved can be simplified to: formation of gallium oxide and arsenic vapor in the water-containing hydrogen stream, followed by reduction of the oxide at the substrate where gallium arsenide is re-formed. Successful growth is possible only by using hydrogen as the carrier gas. Nitrogen is used to purge the furnace, but does not produce an acceptable growth. The growth rate can be controlled both by the temperature of the water reservoir and the rate of hydrogen flow through it.

The material used as a gallium-arsenide source determines the impurity density in the grown layer. If sufficiently pure source material can be obtained, the n^- layer of the rectifier could be grown in the water vapor transport system and the p^+ layer grown in the hydride system. The growth rate in the water system is lower than that of the hydride system and could be a limiting condition.

A photomicrograph of one of the first depositions by the water vapor transport system is shown in Figure 7. Information regarding thickness, growth

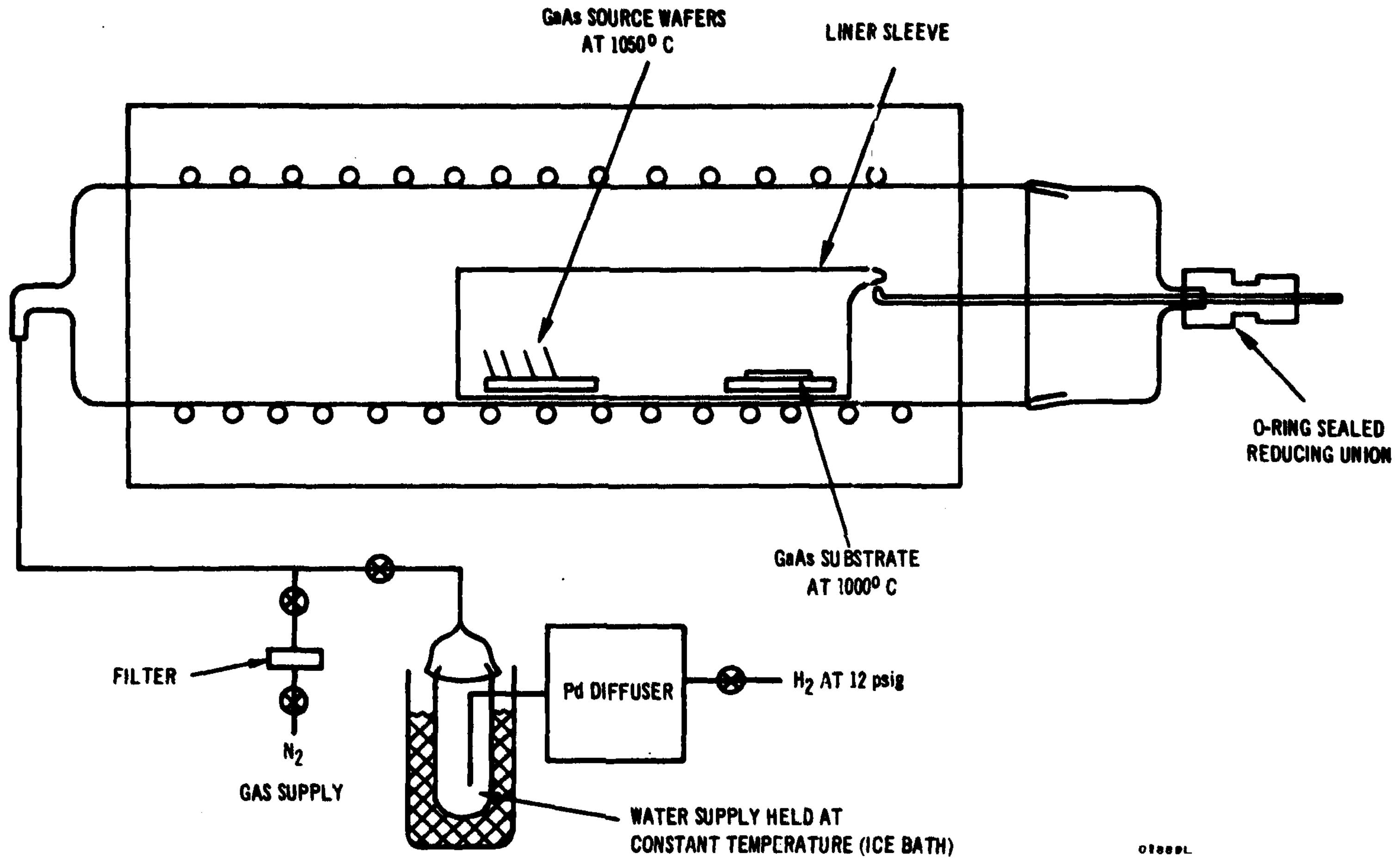


Figure 6. Water Vapor Transport GaAs Epitaxy System

A. STRUCTURE OBSERVED ON PORTION OF WATER TRANSPORT EPITAXIAL GROWTH (X160)

B. A MORE REPRESENTATIVE PORTION OF THE GROWTH DEPICTED ABOVE (X160)

Figure 7. Photomicrographs of One of the First Depositions by the Water Vapor Transport System

rate, and carrier concentration has not been obtained for this growth yet. The square figures are characteristic of the $\{100\}$ surface.

2. VAPOR HYDRIDE TRANSPORT SYSTEM

The vapor hydride transport system has been described elsewhere[2,4], and no basic changes in the operating mechanism have been introduced. However, a number of changes designed to reduce the possibility of contamination during growth have been introduced. These changes involve protection from outside leaks and prevention of corrosion caused by the hydrogen chloride gas used in the process.

In order to minimize leaks and corrosion in the system, the valves and fittings used have been changed to helium-leak tested versions which expose only teflon surfaces to the gas stream. The gas lines and manifolds are constructed exclusively of glass, and a special metering valve of hydrogen-chloride-resistant Hastelloy C has been ordered for the hydrogen chloride delivery system.

To eliminate leaks at the points where push rods must enter the growth tube, quartz trubore bearings have been eliminated and O-ring sealed teflon fittings which permit push-rod motion have been substituted.

Complete assembly of the system is expected shortly.

SECTION IV
PROGRAM FOR NEXT INTERVAL

The materials work will consist of epitaxial growth in both the water vapor transport and, when completed, the vapor hydride transport system. The structural properties of films and the influence of substrate parameters on these structural properties will be examined using the water transport system. The electrical properties of the films will be examined by evaporating Schottky barrier diodes on the films. P-N junctions will be grown by the vapor hydride transport system and evaluated structurally and electrically.

Improved ohmic contacts to gallium arsenide will be investigated. An experimental design for an improved high-temperature package will be developed.

SECTION V
REFERENCES

1. Abrahms and Buiocchi, J. App. Phys. 36, 2855, (1965)

2. Technical Report AFAPL-TR-68-2, Air Force Aero Propulsion Laboratory, Wright-Patterson Air Force Base, Ohio

3. Lawley, K. L., J. Electrochem. Soc. 113, 1, (1966).

4. Tietjen and Amick, ibid., 724.

UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) RCA Electronic Components Somerville, New Jersey	2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED 2b. GROUP

3. REPORT TITLE

300°C Transformer Rectifier

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Interim Technical Report No. 1, 1 January 1968 to 31 March 1968

5. AUTHOR(S) (Last name, first name, initial)

Krassner, L.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
April 1968	21	4

8a. CONTRACT OR GRANT NO. F33615-68-C-1245 new b. PROJECT NO. 3145 c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. AVAILABILITY/LIMITATION NOTICES

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Aero Propulsion Laboratory APIE-3.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY Air Force Aero Propulsion Laboratory APIE-3 Air Force Systems Command Wright-Patterson Air Force Base, Ohio

13. ABSTRACT

Techniques for preparing gallium-arsenide surfaces free of damage and suitable for epitaxial growth were ~~have been~~ investigated. A method of substrate preparation to be used for preliminary growth studies was ~~has been~~ introduced. Growth studies ~~have been~~ started in a water vapor transport epitaxial system. This system ~~has been~~ constructed specifically to evaluate the structure of epitaxial films in relation to the substrate. Design improvements to reduce contamination of epitaxial layers due to leaks and corrosion ~~have been~~ made on the vapor hydride epitaxial system. This improved system is expected to be in operation shortly.

DD FORM 1 JAN 64 1473

UNCLASSIFIED
Security Classification